

## TITLE: ANALYTICAL METHODS FOR TOXINS

**MILESTONE SHC 2.5.1:** Provide one forecast on the occurrence, distribution, and frequency of a harmful algal bloom in the U.S. and develop capability to provide future forecasts.

**CCEHBR SCIENTISTS/INVESTIGATORS:** Peter Moeller (PI), Mark Busman, Robert Roberts, Kevin Beauchesne, Stephen Eaker, John S. Ramsdell

**EXTERNAL COLLABORATORS:** U.S. FDA, EPA, National Research Council, Mote Marine Laboratory

**OBJECTIVES OF RESEARCH ACTIVITIES:** To apply molecular structure-based chemical and physical technologies (e.g., nuclear magnetic resonance spectroscopy, NMR, mass spectrometry, MS) in efforts to unambiguously identify biotoxins contained in any sample matrix.

**DESCRIPTION OF RESEARCH ACTIVITIES:** These research activities are focused on the development of fundamental ultra-sensitive and unambiguous identification and detection of marine toxins. The products of this work provide the capability of providing unequivocal identification of the molecule(s) responsible for toxic activity. Such information is critical in determining the cause of mortalities or illness, as well as in determining the appropriate remedial response. These analytical methods also provide the ability to work with the many sample matrices involved in any given toxic event.

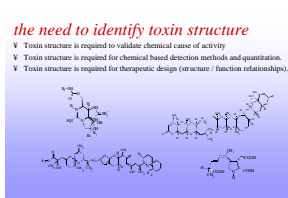
We couple the accuracy and sensitivity of NMR and MS data to that derived from bioassay screening protocols to yield highly reliable identification and quantification of marine toxins. We are developing and applying such methods for important marine toxins such as the saxitoxins, ciguatoxins, brevetoxins, maitotoxins, yessotoxin, okadaic acid, domoic acid, palytoxin and *Pfiesteria* toxin.

## Graphic/Image/Figures

NMR



Structure



LC-MS/MS



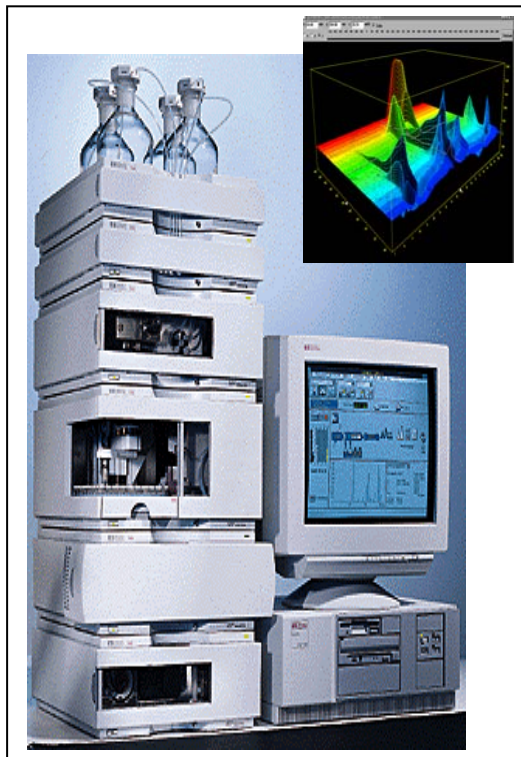
## Selected Highlights

\* Analytical measurements using NMR, HPLC and LC-MS have resulted in the detection and initial characterization of two new toxins

\* NMR and MS of a water soluble toxin derived from *Pfiesteria piscicida*

\* HPLC, NMR and LC-MS of a novel water soluble toxin derived from *Trichodesmium thiebauti*.

\* In addition, deuteration of brevetoxin-2 was completed via collaborative studies with the EPA as the first step in developing ultra – sensitive detection methodologies for brevetoxin metabolisms monitoring.



## Presentations

**Leighfield T.A.**, Van Dolah F.M., Doucette, G.J., Moeller, P.D. Morton, S.L., Wiggins, N., and Ramsdell, J.S.. NOAA Marine Biotoxins Analytical Response Team. Xth International Conference on Harmful Algae, 2002, St. Petersburg, FL.

**Wiggins-Mitchell, N.**, P.D.R. Moeller, M. Busman, T. Villareal, B. Richardson, K. Steidinger, and S.L. Morton. Brevetoxin-2 and –3 variability from Strains of *Karenia brevis* isolated from Florida and Texas using tandem mass spectrometry. Xth International Conference on Harmful Algae, 2002, St. Petersburg, FL.

**Morton, S.L.**, T. Villareal, S. Eaker, M. Hsia, T. Schock, and P.D.R. Moeller. Algicidal activity of a water-soluble compound extracted from *Trichodesmium thiebauti*. Xth International Conference on Harmful Algae, 2002, St. Petersburg, FL.

